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TRANSMITTANCE MEASUREMENTS OF OPTICAL MATERIALS AS AFFECTED BY WEDGE ANGLE AND REFRACTIVE INDEX IN THE 2- TO 15-MICRON RANGE

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By

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Aviation Ordnance Department

MAY 14 1963

ABSTRACT. Error in the measurement of transmittance can be made with samples having nonparallel faces. This has been investigated because of the extensive use of transmittance as a tool for determining properties of semitransparent materials. Specimens of fuzed quartz, arsenic trisulfide, silicon, and germanium, representing a range of refractive indices, were fabricated to provide 0-, 1-, 2-, 5-, and 10-milliradian wedges across 1-inch diameter disks. Transmittance measurements were taken in the 2- to 15-micron range with Perkin-Elmer Models 21 and 221 spectrophotometers, and at 2 microns with a Cary Model 14 spectrophotometer, and correlated with refractive index and wedge angle. For materials with a low refractive index, such as fused quartz, the error introduced even by the 10-mil wedge was negligible. However, for high index materials, such as germanium, as much as 60% error was introduced by the 10-mil wedge.

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U.S. NAVAL ORDNANCE TEST STATION

China Lake, California

April 1963

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C. BLENMAN, JR., CAPT., USN
Commander

WM. B. McLEAN, PH.D.
Technical Director

FOREWORD

This report describes work accomplished during the period July 1961 to July 1962 on the effect of non-plane-parallel material on spectrophotometer transmittance measurements in support of the Infrared Detection Program at the Naval Ordnance Test Station, China Lake, California. This investigation was made for the Bureau of Naval Weapons under WepTask RMWC-43-001/216-1/F001-05-01.

This report has been reviewed for technical accuracy by H. P. Leet and H. E. Bennett.

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Under the authority of
WM. B. McLEAN
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INTRODUCTION

Past irreproducibility of transmittance spectra of various optical materials has led to an investigation of the magnitude of the error possible in transmittance measurements if the sample faces are not parallel. Transmittance measurement is a fundamental tool used at present to determine the optical properties of semitransparent materials. Determining band structure, degree of doping in semiconductors, and optical constants of materials exemplify some of the uses for transmittance measurements. Therefore, it is important to identify and, if possible, remove sources of systematic error in transmittance measurements.

This study was made to investigate the problem of transmittance as related to refractive index and to surface parallelism, to ensure accuracy of transmittance measurements.

As a method of studying the problem, transmittance measurements on controlled optical specimens were analyzed. Specimens of fused quartz, arsenic trisulfide, silicon, and germanium were prepared with parallel and with controlled nonparallel sides. This report presents the results of these transmittance measurements made with commercially available instruments. The results should be useful as a guide in determining how parallel the sides must be to achieve a given degree of accuracy with these instruments.

THEORY

There are several parameters that must be considered before any analysis of this problem can be meaningful. The angle between the normals to the sides of the specimens will be called the wedge angle, α. A wedge will deflect a beam of light; designate this deflection angle as θ. The deflection angle is a function of the wedge angle and the refractive index, n. The refractive index, in turn, depends upon the material and the wavelength. Further, the transmittance value obtained for a given wedge specimen will vary with the spectrophotometer concerned and with the orientation of the wedge in the spectrophotometer beam (Fig. 1).

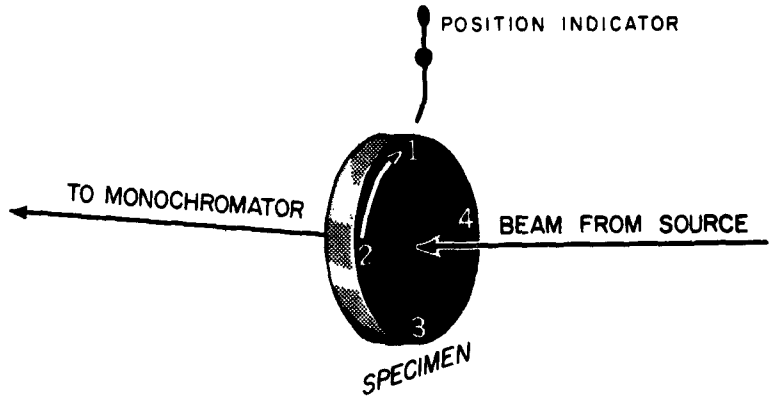

FIG. 1. Diagram Showing Specimen Orientation.

The following is a list of abbreviations and symbols used in this report:

a	Difference in thickness across a 1-inch-diameter specimen
n	Refractive index
R	Reflectance
α	Wedge angle between normals
θ	Beam deflection angle
θ'	Internally reflected beam deflection angle
τ	Transmittance
τ_o	Transmittance of parallel specimen
$\%\tau$	Transmittance, percent
Inst	Spectrophotometer
Pos 1	Thickest part of specimen is upward (0 degree)
Pos 2	Thickest part is rotated 90 degrees clockwise
Pos 3	Thickest part is rotated 180 degrees clockwise
Pos 4	Thickest part is rotated 270 degrees clockwise

The beam deflection angle, θ, must be calculated for each wedge specimen at each wavelength (Fig. 2). The equation used for this purpose is that for a thin prism, $\theta = \alpha(n - 1)$, where α is the prism angle and n is the refractive index. For this application, α is small and can be replaced by tan α, which is equal to a, the difference in thickness across a 1-inch-diameter disk. Consequently, $\theta = a(n - 1)$.

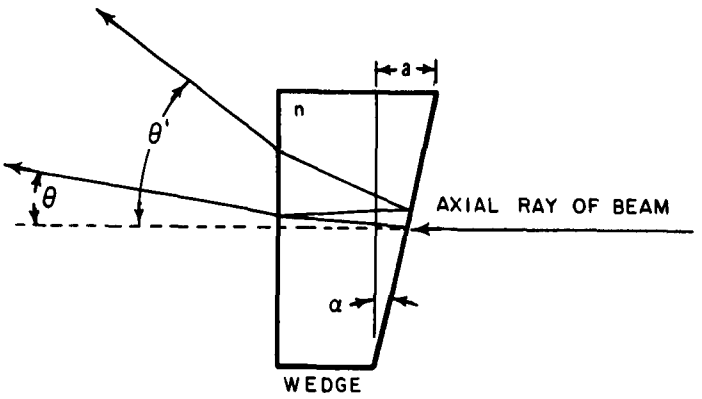

FIG. 2. Detail Diagram Showing Beam Deflection, No Scale.

With the average thickness of the specimens being 1/4 inch, the lateral displacement of the beam is negligible compared to the angular deflection. Furthermore, with small angles involved and no optically active materials, the difficulties that might be introduced because of polarization are negligible.

PROCEDURE

The materials employed in this study were fused quartz, arsenic trisulfide, silicon, and germanium. The choice of materials was ruled by physical properties with special emphasis on the range of refractive indices 1.5 to 4. The specimens were disks 1 inch in diameter and approximately 1/4-inch thick, and were fabricated to provide 0-, 1-, 2-, 5-, and 10-milliradian (mil) wedges. The faces were polished optically flat to within 5 green mercury fringes, and were controlled with respect to the wedge angle between them.

The wedge angles of the specimens were measured on a prism spectrometer equipped with a Gauss eyepiece. The reproducibility was determined by repeated readings to be ± 0.00006 radian. Below is a table showing the wedge angles of the specimens. The wedge angle, in radians, is equal to the difference in thickness, in inches, across a 1-inch disk.

TABLE 1. Wedge Angles of Specimens, in Radians

Material	Plane-parallel	0.001-rad wedge	0.002-rad wedge	0.005-rad wedge	0.010-rad wedge
Quartz	0.00027	0.00108	0.00187	0.00504	0.01008
Arsenic trisulfide	0.00001	0.00113	0.00219	0.00497	0.01002
Silicon	0.00028	0.00121	0.00201	0.00512	0.01016
Germanium	0.00008	0.00118	0.00217	0.00526	0.01014

All disks of the same type material were cut from a common single crystal, or from the same supply, eliminating possible optical variations between them. The specimens used were a waterfree fused quartz, Type 106, from General Electric, an arsenic trisulfide from Barr and Stroud, and silicon and germanium blanks from Knapic Electro-Physics, Inc. The impurity of the silicon was given as approximately 6 parts per billion (ppb) borron and approximately 20 ppb phosphorus; that for germanium was approximately 1 ppb total impurities. Specimens were prepared by the John H. Ransom Laboratories.

Three spectrophotometers were used to obtain data for analysis to determine the variation of the possible error in transmittance between types of instruments. The instruments investigated were Perkin-Elmer Models 21 and 221, and a Cary Model 14. In principle, these are all dual-beam instruments. The Perkin-Elmer Models 21 and 221 are optically alike. This is advantageous for showing variations in identical instruments. Diagrams of the optical systems of these spectrophotometers are shown in Fig. 3 and 4.

Two precision holders were fabricated for retaining the specimens securely and accurately in the spectrophotometers. Figure 5 shows one of these holders. The ring in which the specimen is placed can be rotated with the spectrophotometer beam as the axis. Figure 6 shows the holder in the standard sample mount for the Cary Model 14, and Fig. 7 shows the Perkin-Elmer Model 221 with a specimen holder in place.

Transmittance spectra were taken of each wedge for various rotation positions in the beam. In addition, plots of transmittance versus rotation were taken at constant wavelength for more insight into the effect of wedged specimens. Plots of quartz are not shown because effects from wedge were negligible. (Fig. 8, 9, 10.)

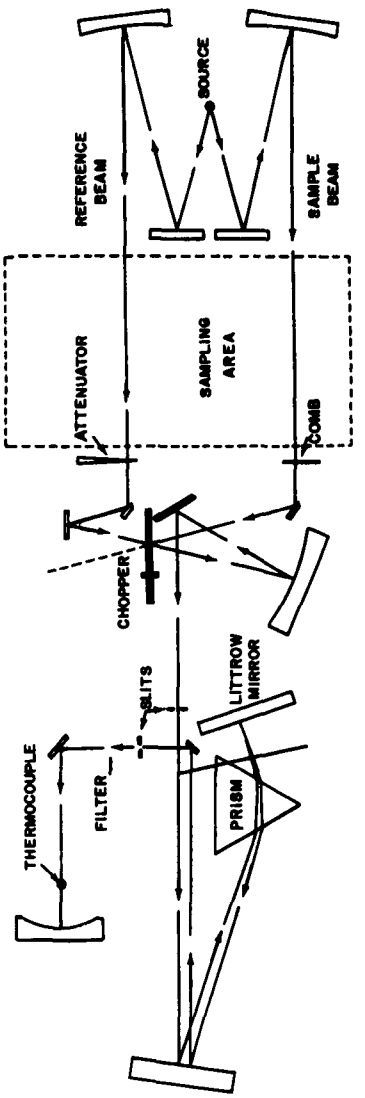

FIG. 3. Optical Diagram of the Perkin-Elmer Models 21 and 221 Spectrophotometers.

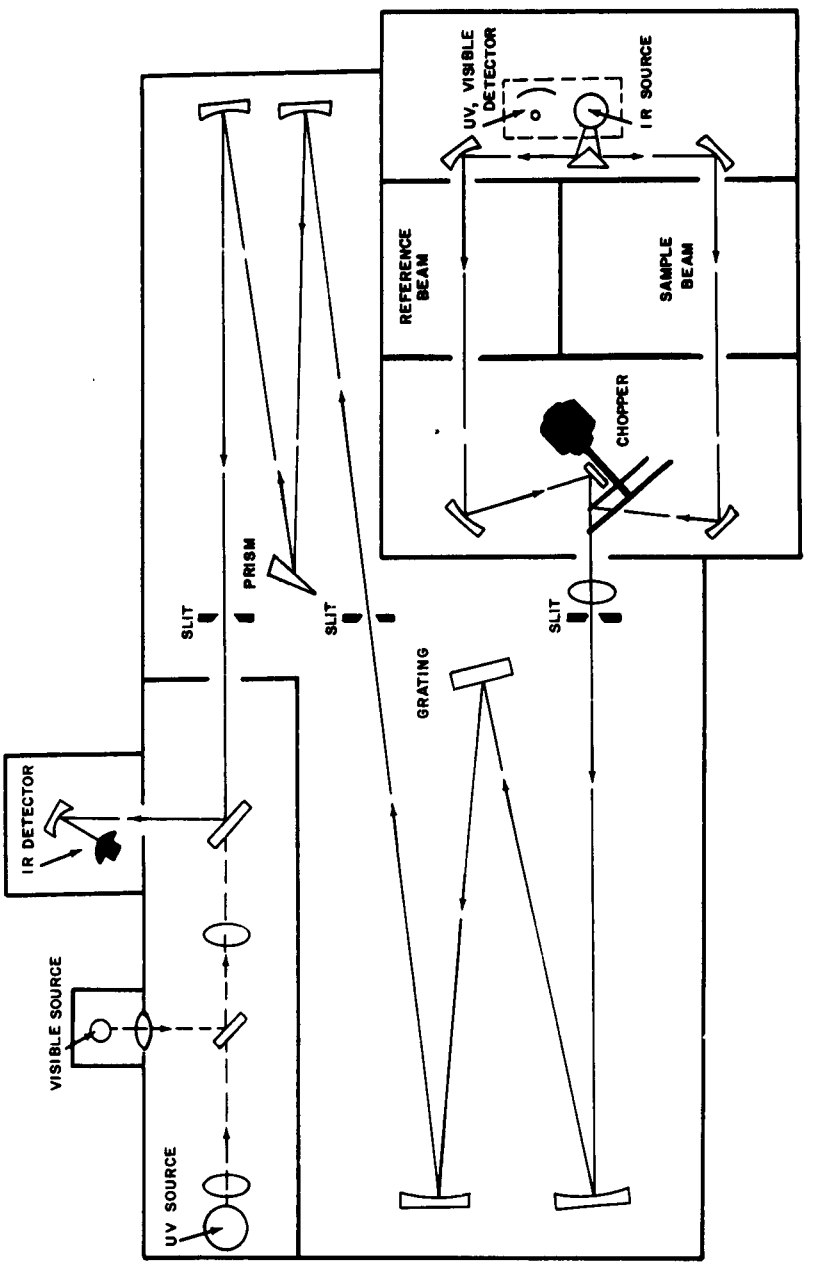

FIG. 4. Optical Diagram of the Cary Model 14 Spectrophotometer.

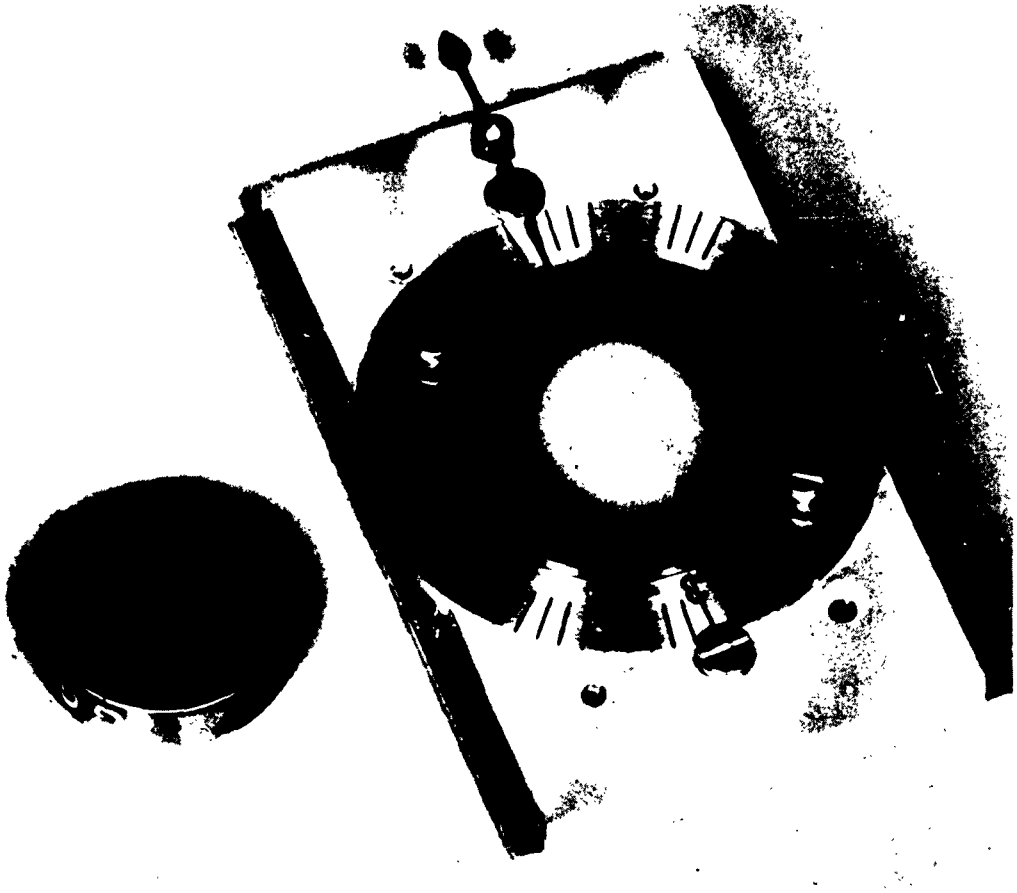

FIG. 5. Specimen Holder and Specimen.

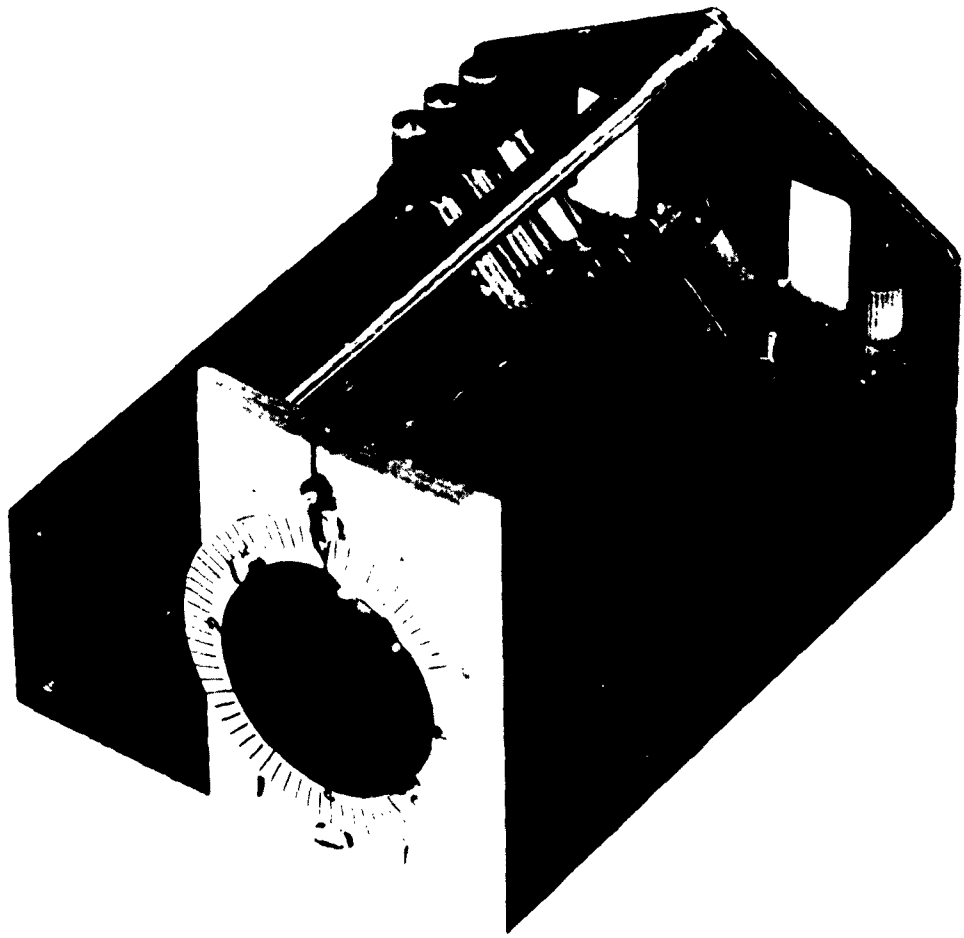

FIG. 6. Specimen Holder Mounted for Use in the Cary Model 14 Spectrophotometer.

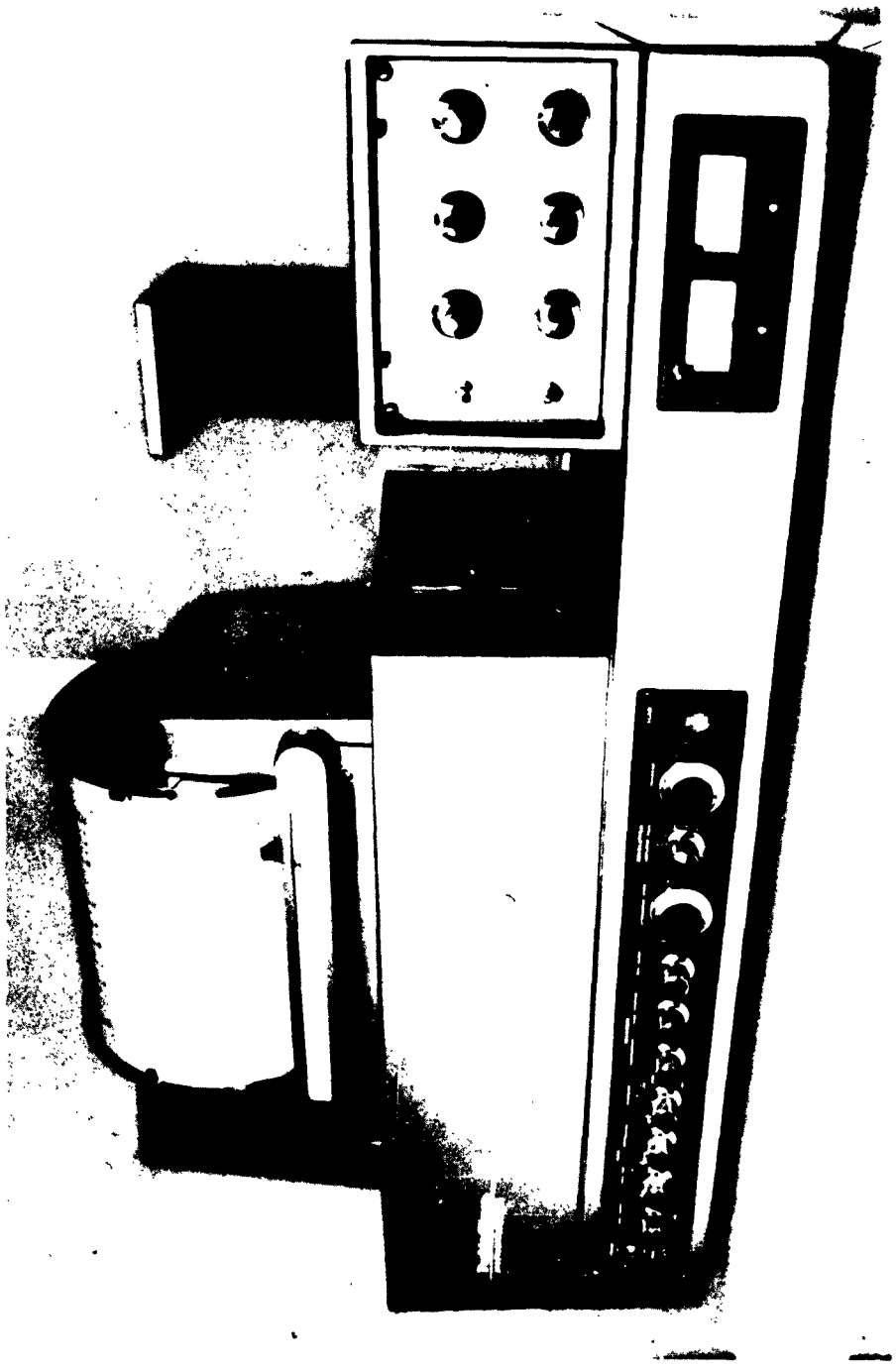

FIG. 7. Perkin-Elmer Model 221 Spectrophotometer with Specimen Holder in Place.

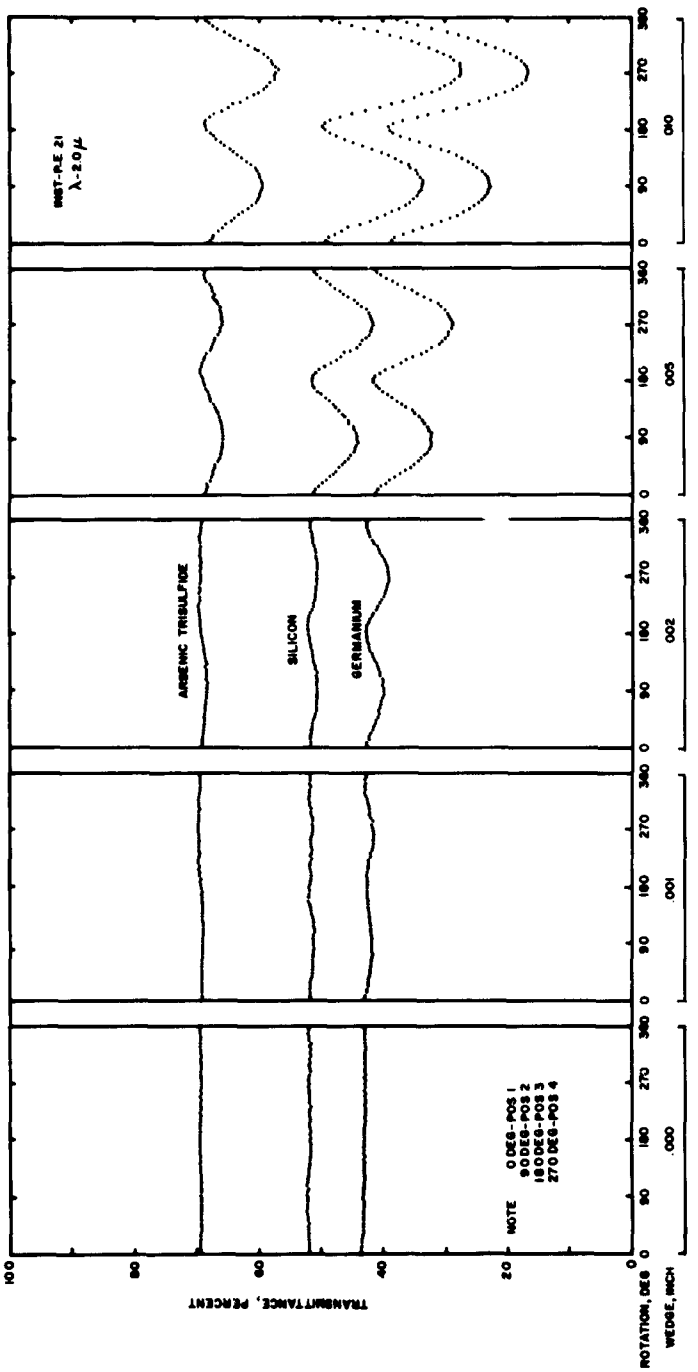

FIG. 8. Percent Transmittance for Specimen Rotation on the Perkin-Elmer Model 21 at 2-Micron Wavelength.

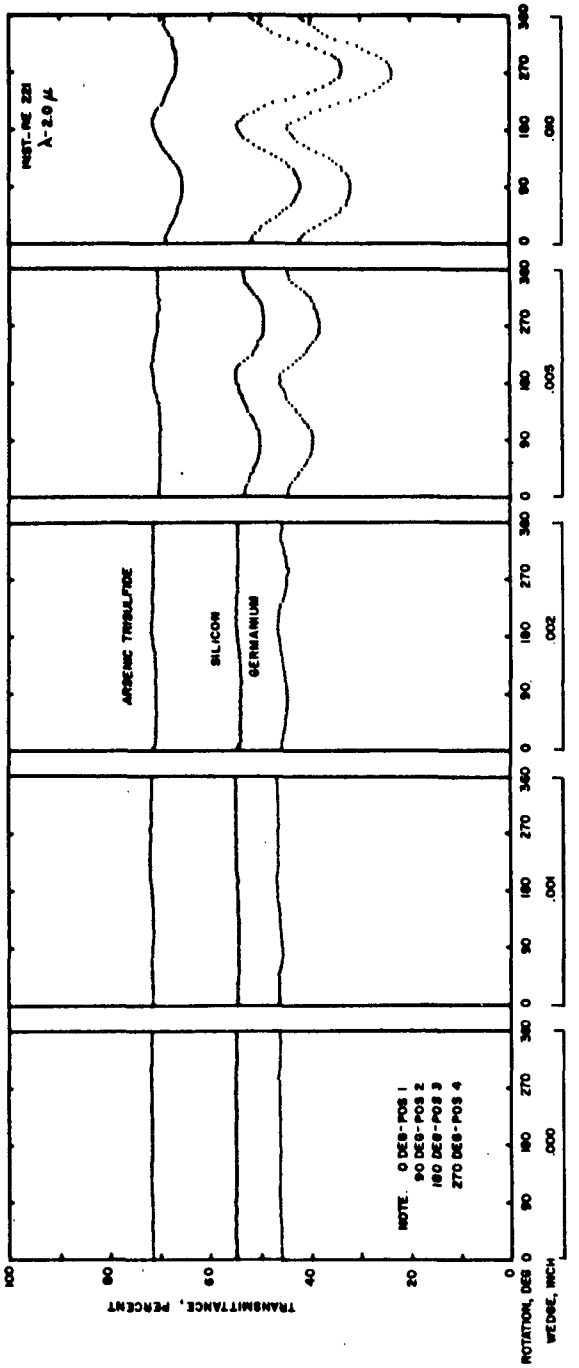

FIG. 9. Percent Transmittance for Specimen Rotation on the Perkin-Elmer Model 221 at 2-Micron Wavelength.

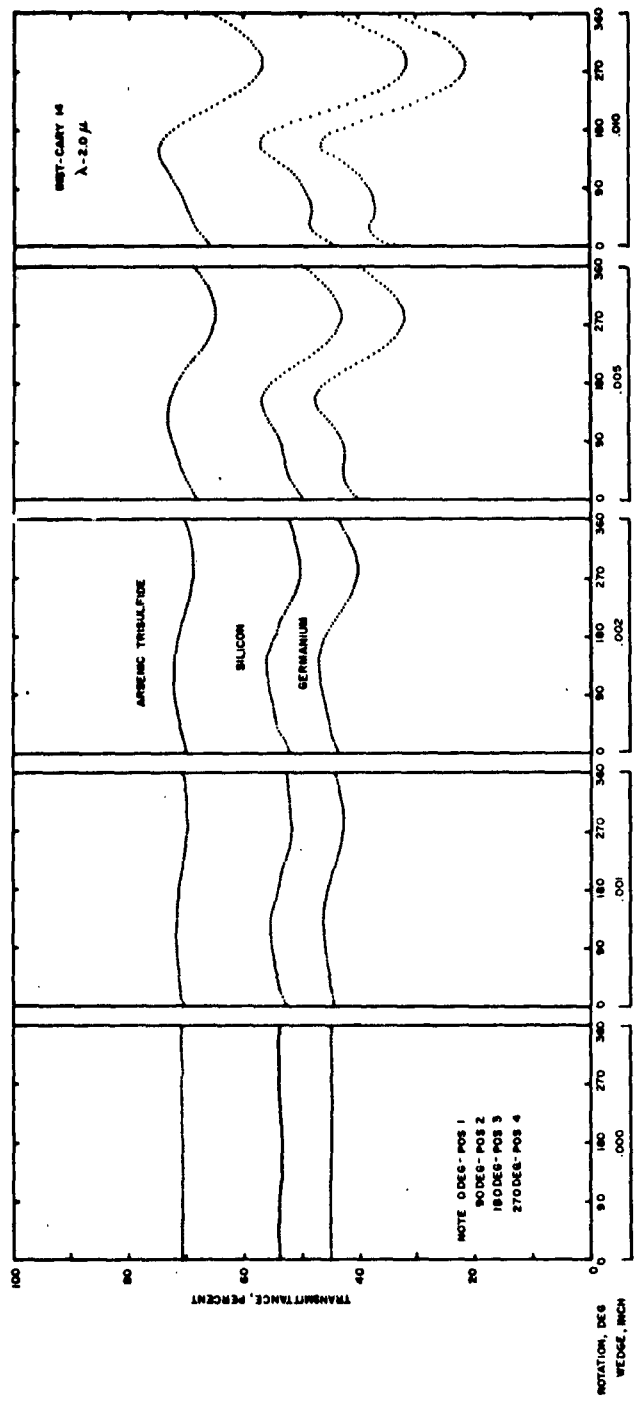

FIG. 10. Percent Transmittance for Specimen Rotation on the Cary Model 14 at 2-Micron Wavelength.

DATA AND ANALYSIS

Samples of transmittance spectra are given in Fig. 11 and 12. Similar spectra were obtained for all specimens of each material. Because the transmittance of each material is different, the values cannot be compared directly. However, they can be compared if they are normalized or are given with respect to the underlected beam, that is, when the transmittance is given relative to the value τ_o, obtained for the parallel specimen. The relative transmittance is plotted against the computed deflection angle for the four orientations of the wedge in the spectrophotometer beam. All values of refractive index used for this computation were taken from the IRIA State-of-the-Art Report on Optical Materials.[1] Since the ordinate is relative to the parallel specimen of each material and the computation of the deflection angle takes into account the refractive index of each material, the relative transmittance plots are not a function of material. Hence, the parameters for these plots are specimen orientation, instrument, and wavelength. The resulting information is displayed in Fig. 13 through 22.

In computing the deflection angle of the spectrophotometer beam for each sample, multiple internal reflections were not included. The portion of the beam that is internally reflected twice is given as $(1 - R)^2 R^2$, where R is reflectance. The portion that is internally reflected four times is given as $(1 - R)^2 R^4$.

At normal incidence, reflectance is given by

$$R = \left[\frac{(n - 1)}{(n + 1)}\right]^2.$$

This expression can be used with high accuracy at nearly normal incidence. At 2-micron wavelength for each material, the contributions to the transmitted beam from the second and fourth internal reflection are given in Table 2.

[1] University of Michigan. Optical Materials for Infrared Instrumentation, by S. S. Ballard, K. A. McCarthy, and W. L. Wolfe. Willow Run Laboratories, Ann Arbor, Michigan, January 1959. (State-of-the-Art Report No. 2389-11-8), UNCLASSIFIED.

TABLE 2. Percentage of Initial Beam from Second and Fourth Internal Reflection

Material	Refractive index	Second, %	Fourth, %
Quartz	1.521	0.17	0.00
Arsenic trisulfide	2.418	2.03	0.06
Silicon	3.453	4.47	0.41
Germanium	4.108	5.44	0.75

The contribution to the error in transmittance by the loss of the portion of the beam that is internally reflected four times is considered negligible, being less than 1%. However, the portion lost from the twice-reflected beam does contribute significantly to additional energy loss. The deflection for the twice internally reflected portion for a small wedge is given by $\theta' = \alpha(3n - 1)$.

For the worst error condition possible on the materials employed in this study, consider the germanium specimen with a wedge of 10 mils.

$$\theta' = 0.01\left[(3)(4.108) - 1\right] = 0.1132 \text{ rad} \cong 6.5 \text{ degrees}$$

In the most extreme case, specifically left deflection with the Cary Model 14 spectrophotometer, the 5.4% of the initial beam energy twice-reflected is almost entirely lost as a result of the 6.5-degree deflection (Fig. 17). With the germanium transmittance of approximately 50%, the error in transmittance due to multiple internal reflections is, at most, 11% of the true transmittance value.

The 11% error found in the previous paragraph is for extreme error conditions. Consider the magnitude of error caused by multiple internal reflections in a region of more importance; namely, the region in which the relative transmittance is no less than 0.9. This region is spanned by the germanium specimen with a wedge of 2 mils. The deflection angle of the twice internally reflected beam for this 2-mil wedge is 0.023 radian or 1.3 degree. Under the conditions just stated, the 5.4% of the initial beam energy that is deflected 1.3 degree is reduced by no more than 50%. Consequently, the maximum error introduced by multiple internal reflections is 5.4% of the true transmittance. For materials with smaller refractive index, the error will be considerably less than 5.4%.

Instrumental uncertainty is shown by line segments constructed above and below each point in Fig. 13 through 22. These were determined from a precision of ±0.52 transmittance. This uncertainty applies to the three spectrophotometers employed, and has been established from previous experience with these instruments. From these plots, it is obvious that the error introduced by a wedge specimen can be greater than the instrumental uncertainty even for small wedge angles.

It is apparent from Fig. 21 and 22 that the relative transmittance is nearly independent of wavelength. The deflection angle was computed with the appropriate value of refractive index at each wavelength. The implication here is that the results of Fig. 13 through 20 are valid over a large wavelength region.

The possibility of a wavelength shift in the monochromator portion of the spectrophotometer was investigated. A wavelength calibration check was made using a polystyrene film. The check was made with and without a specimen to deflect the beam. No wavelength shift was observed.

With the Perkin-Elmer Models 21 and 221, the specimen holder is in thermal contact with the instrument case. The monochromator is kept at a slightly elevated temperature for stability. It was observed that as the germanium specimens warmed from contact with the monochromator wall, the transmittance at the 2-micron wavelength decreased. This occurred only on the absorption edge. This behavior is explained by the absorption edge shift due to change in temperature.[2] From ambient to monochromator wall temperature there was an 8-centigrade-degree increase. To guarantee reproducibility, the germanium specimens were allowed to reach equilibrium temperature before data were taken.

Another potential complication is the dependence of refractive index on temperature. If this were large it would affect the values of deflection angles; however, this effect is small. For example, the change in index per change in temperature for germanium at 24.5°C and 2-micron wavelength is approximately $5.9 \times 10^{-4}(\text{C deg})^{-1}$. [3]

[2] Fan, H. Y., "Temperature Dependence of the Energy Gap in Semiconductors," PHYS REV, Vol. 82, 1951, p. 900.

[3] Rank, D. H., H. E. Bennett, and D. C. Cronemeyer, "The Index of Refraction of Germanium Measured by an Interference Method," OPT SOC AM, J, Vol. 44, 1954, p. 13.

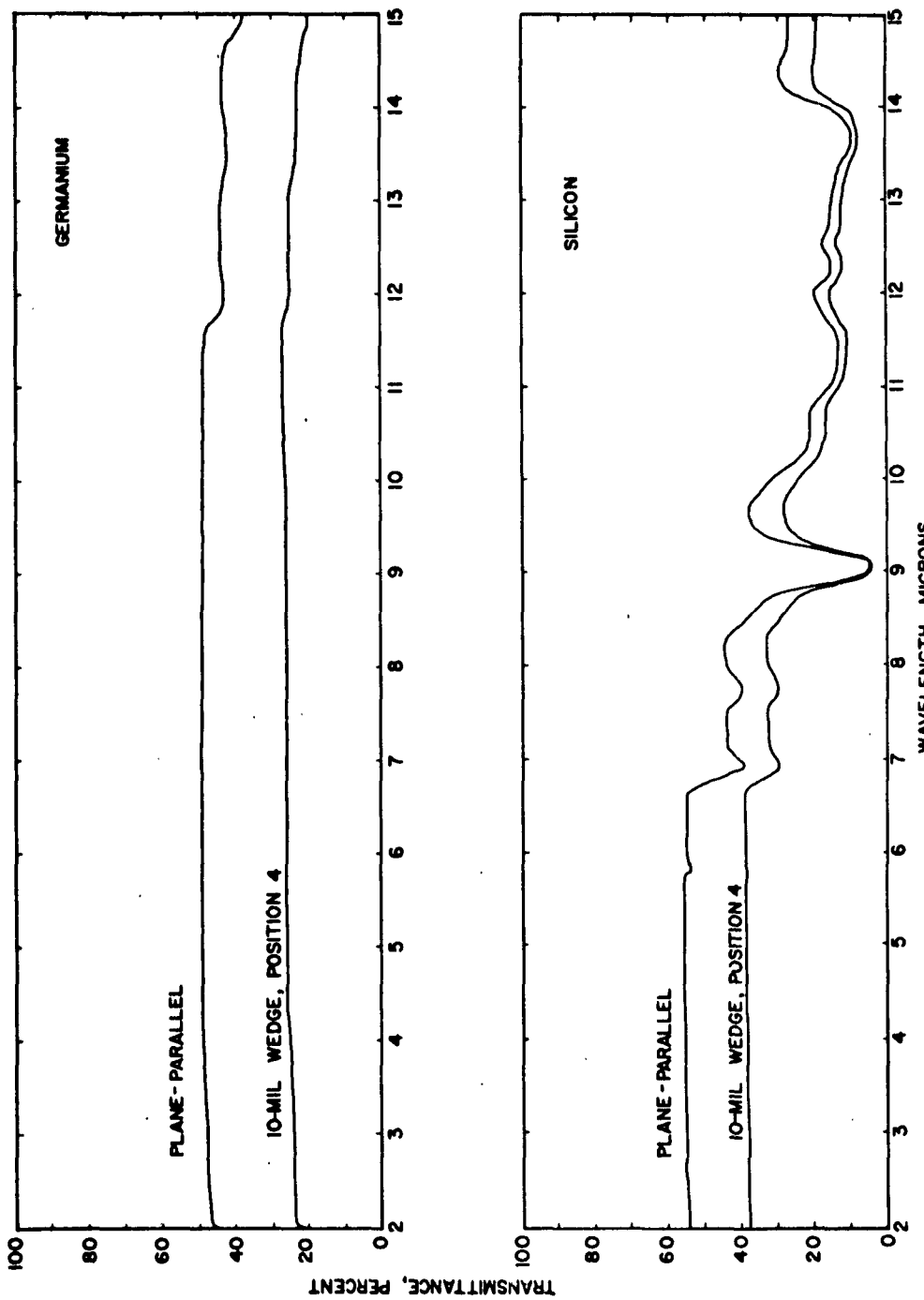

FIG. 11. Transmittance Spectra for Arsenic Trisulfide and Quartz with Plane-Parallel and 10-Mil Wedge Specimens.

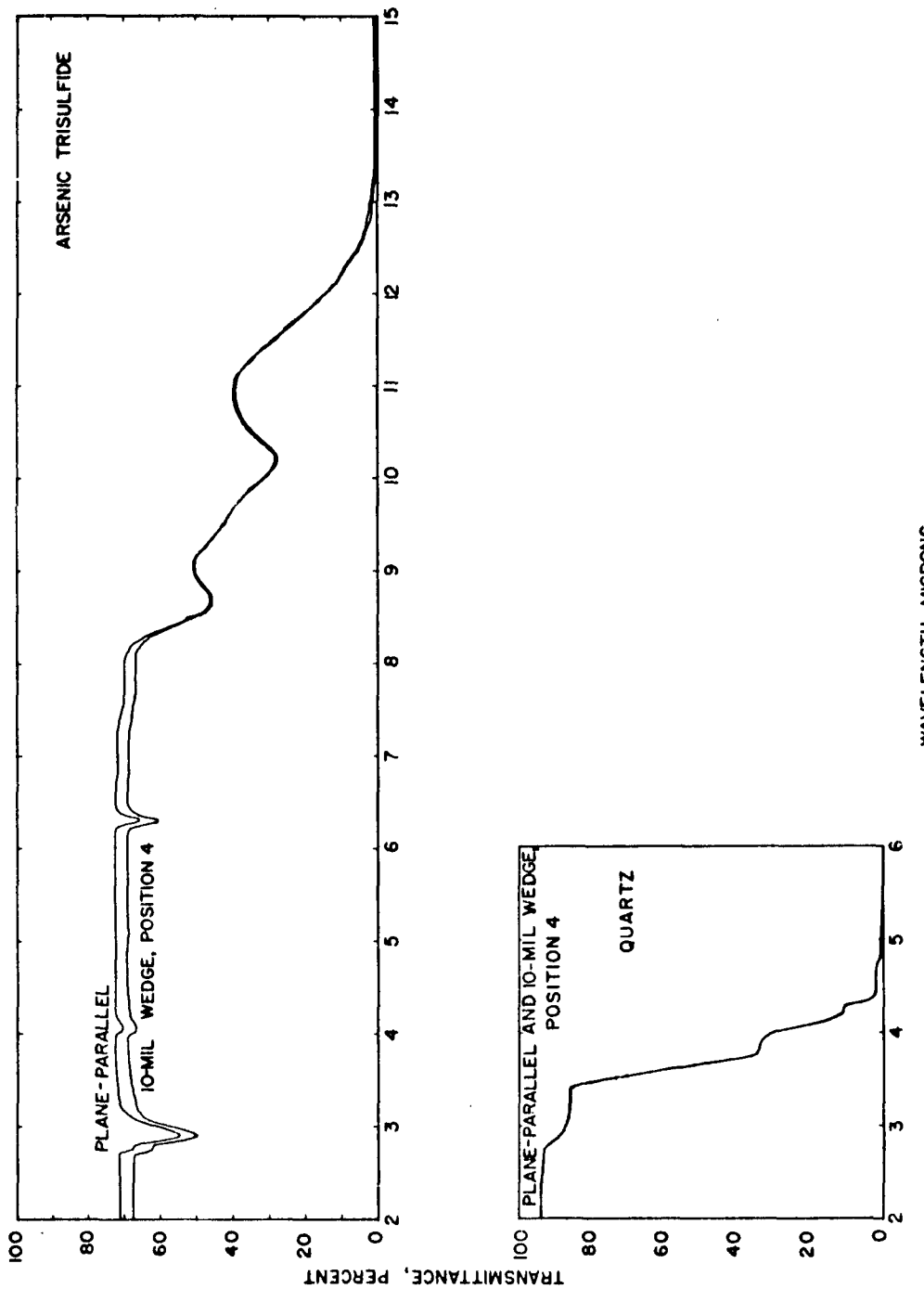

FIG. 12. Transmittance Spectra for Germanium and Silicon with Plane-Parallel and 10-Mil Wedge Specimens.

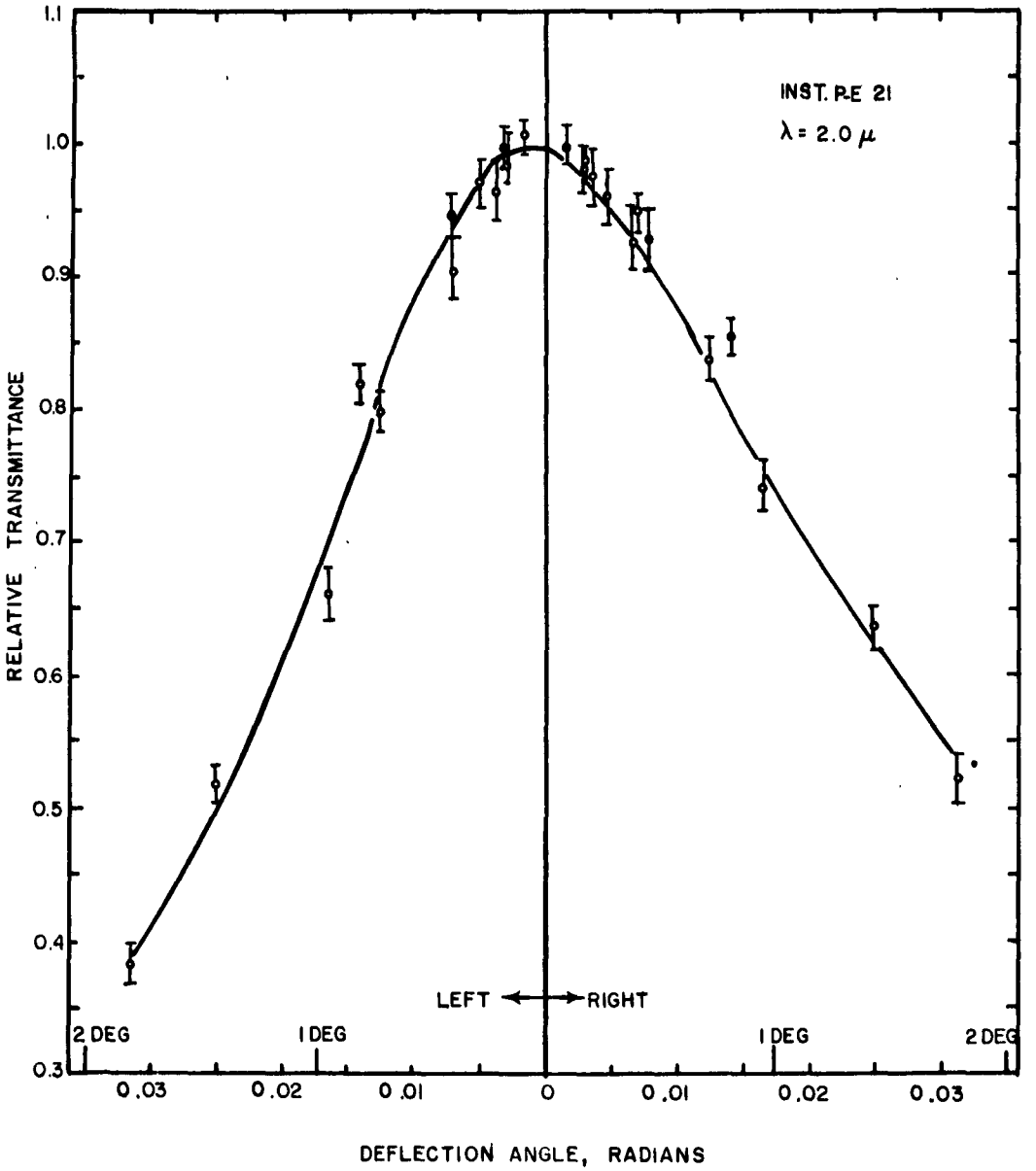

FIG. 13. Relative Transmittance Versus Deflection Angle for the Perkin-Elmer Model 21 at 2-Micron Wavelength, Left-Right Deflection.

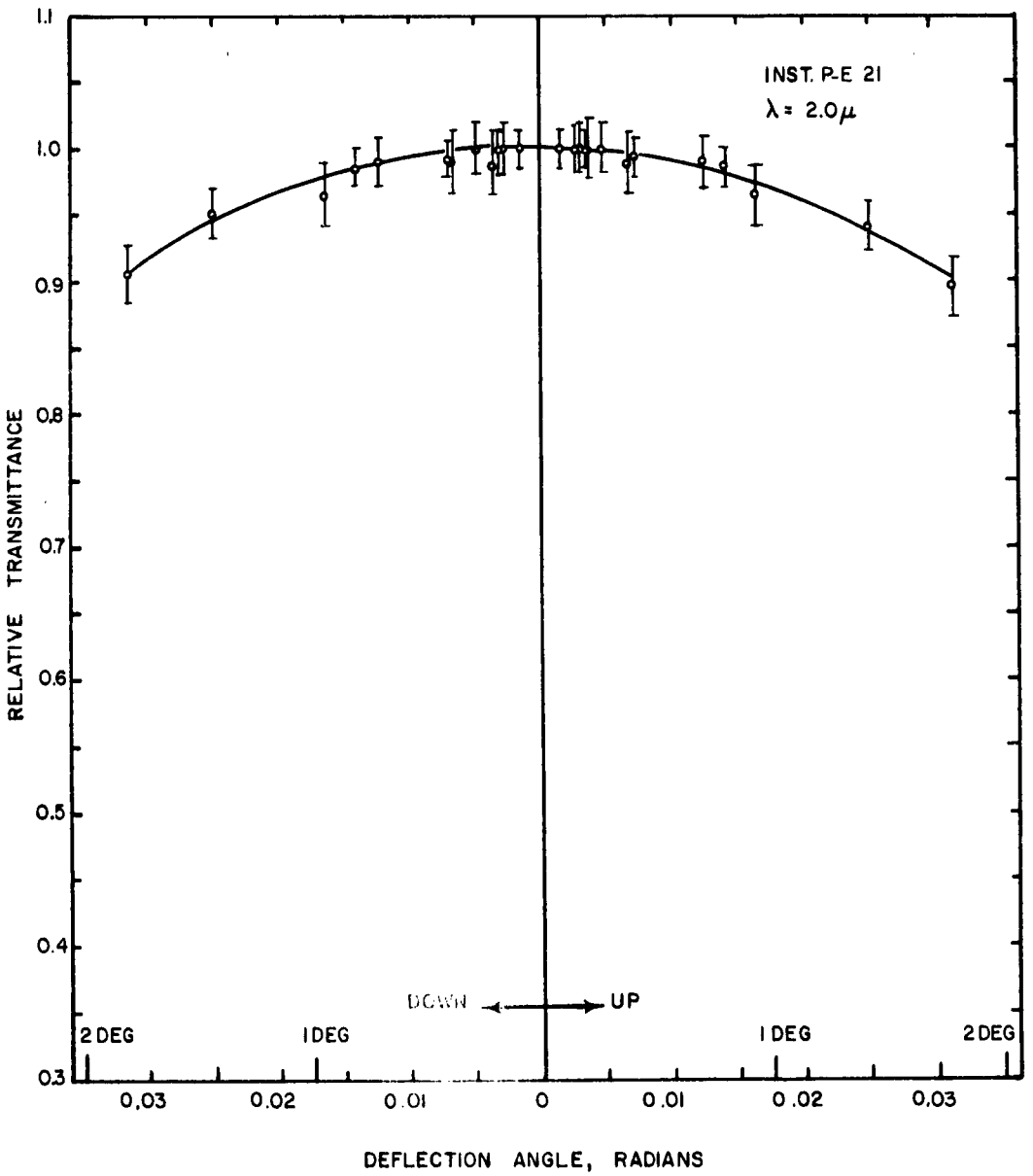

FIG. 14. Relative Transmittance Versus Deflection Angle for the Perkin-Elmer Model 21 at 2-Micron Wavelength, Up-Down Deflection.

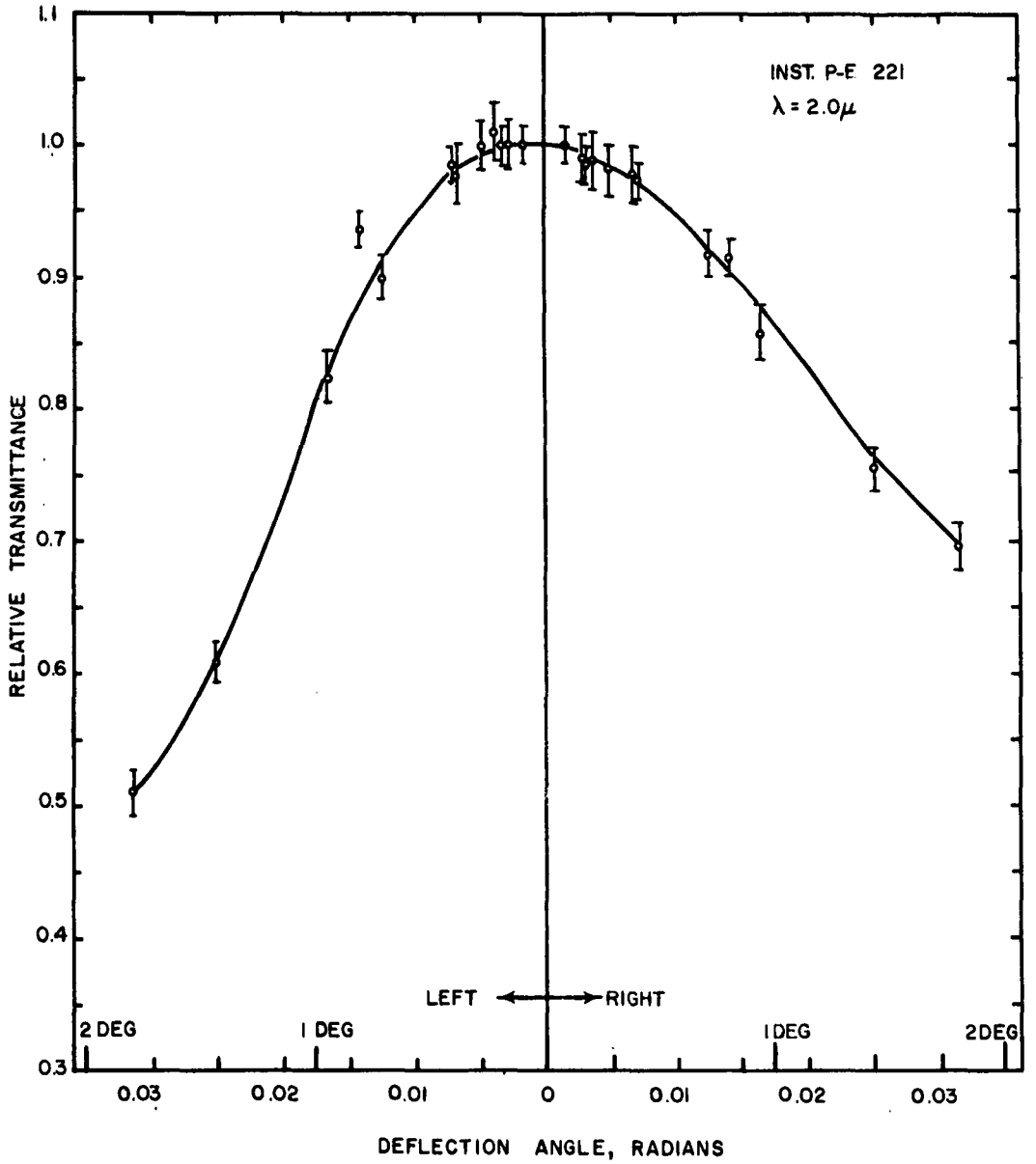

FIG. 15. Relative Transmittance Versus Deflection Angle for the Perkin-Elmer Model 221 at 2-Micron Wavelength, Left-Right Deflection.

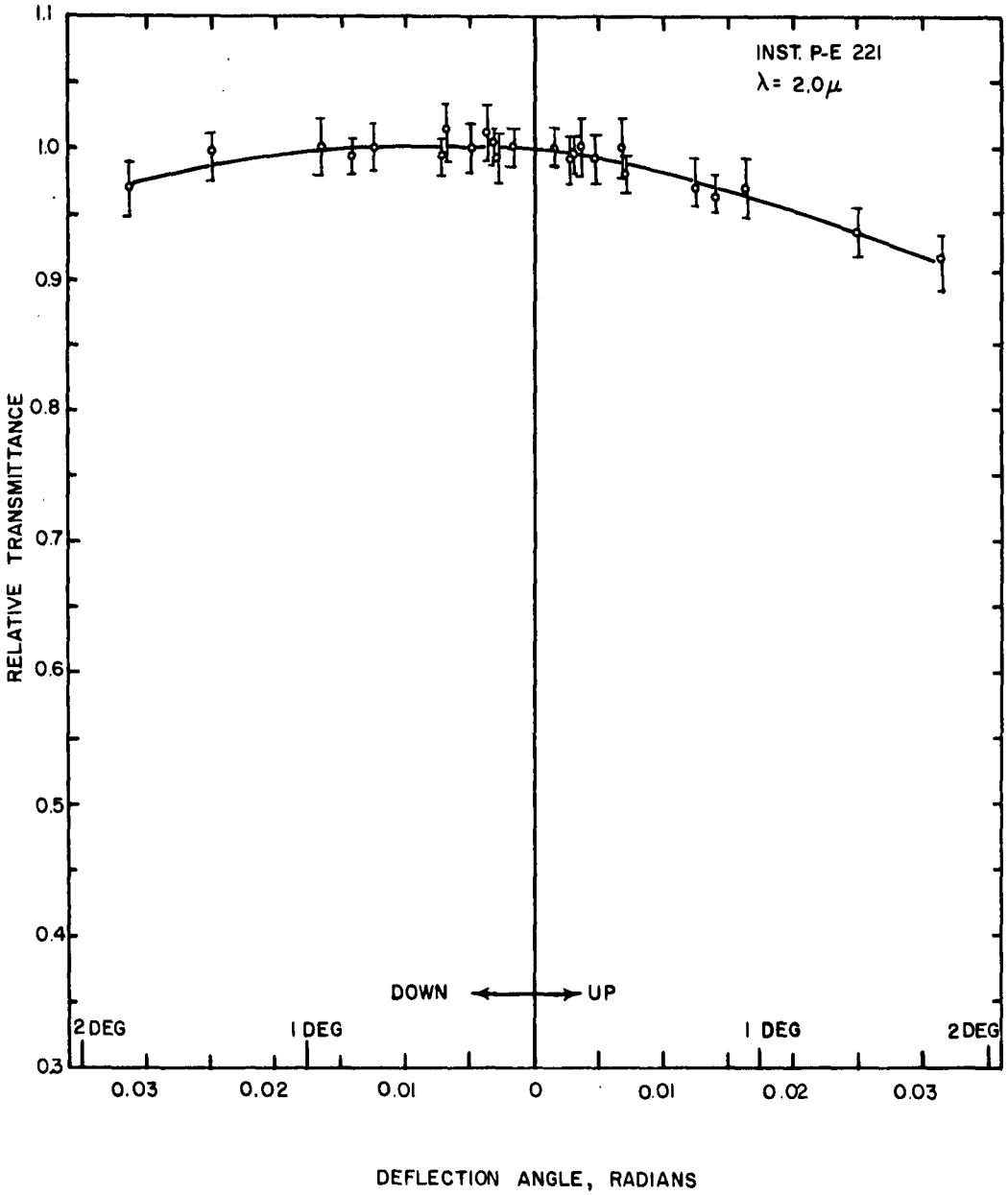

FIG. 16. Relative Transmittance Versus Deflection Angle for the Perkin-Elmer Model 221 at 2-Micron Wavelength, Up-Down Deflection.

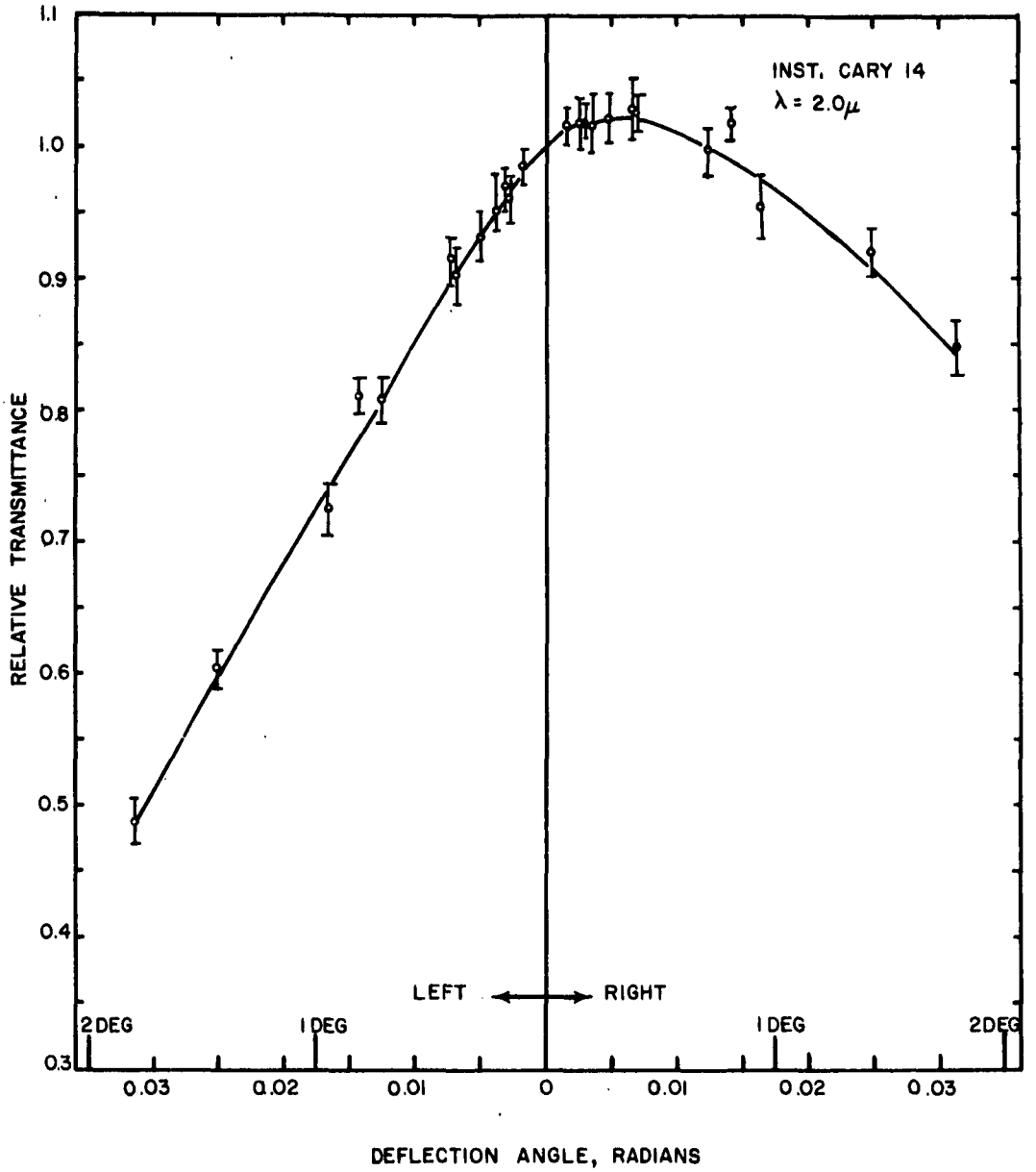

FIG. 17. Relative Transmittance Versus Deflection Angle for the Cary Model 14 at 2-Micron Wavelength, Left-Right Deflection.

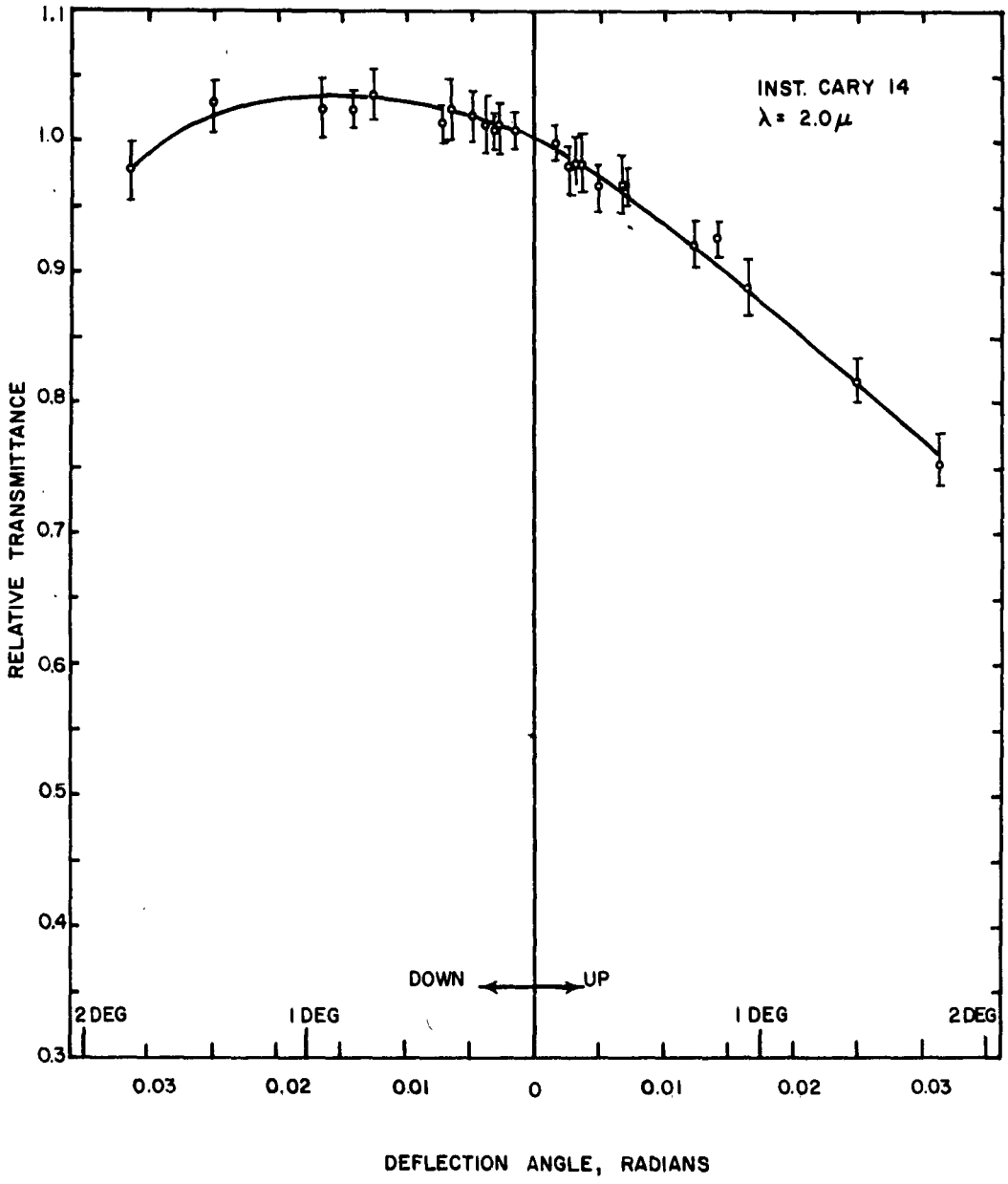

FIG. 18. Relative Transmittance Versus Deflection Angle for the Cary Model 14 at 2-Micron Wavelength, Up-Down Deflection.

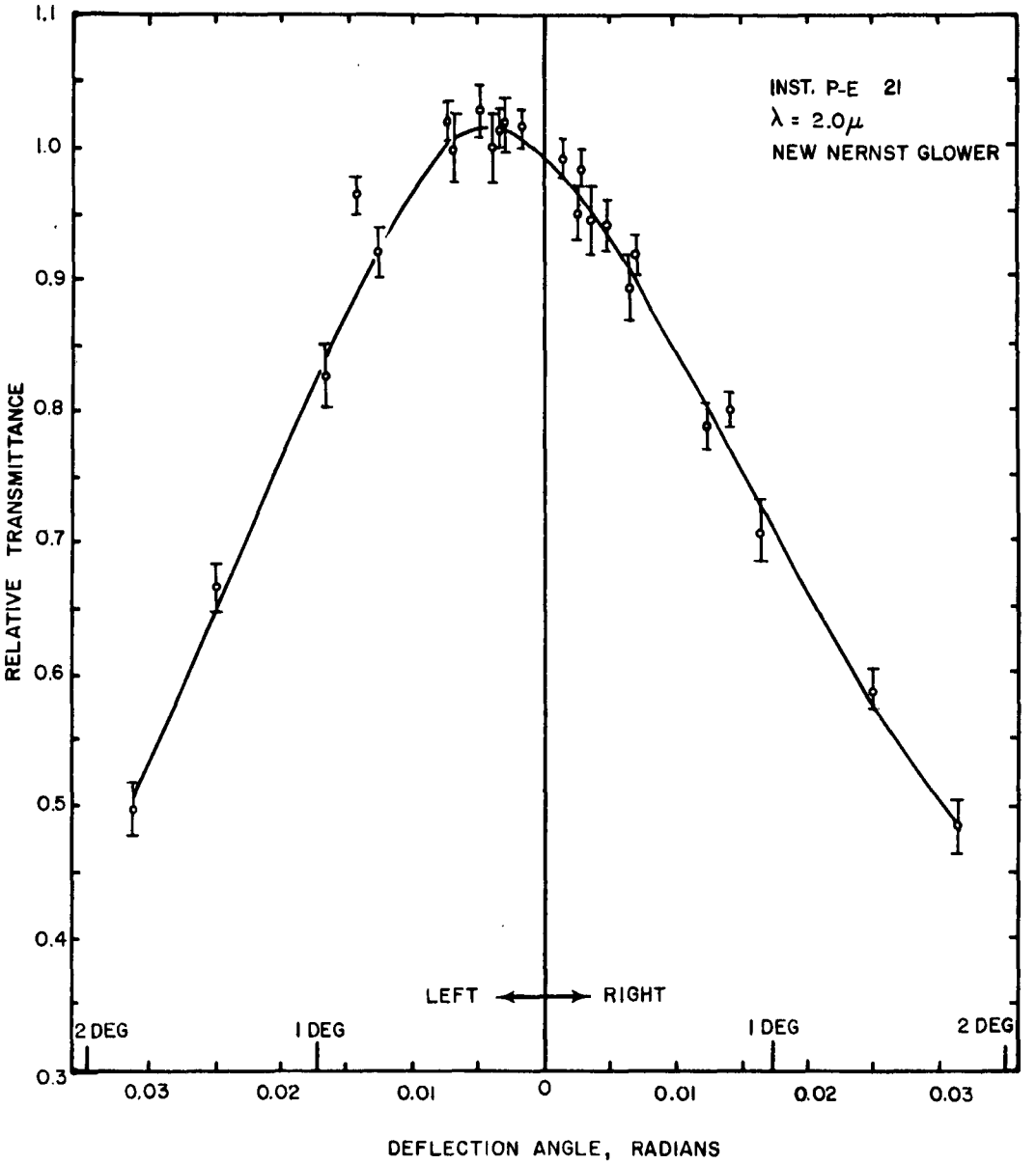

FIG. 19. Relative Transmittance Versus Deflection Angle for the Perkin-Elmer Model 21 at 2-Micron Wavelength with Newly Installed Nernst Glower, Left-Right Deflection.

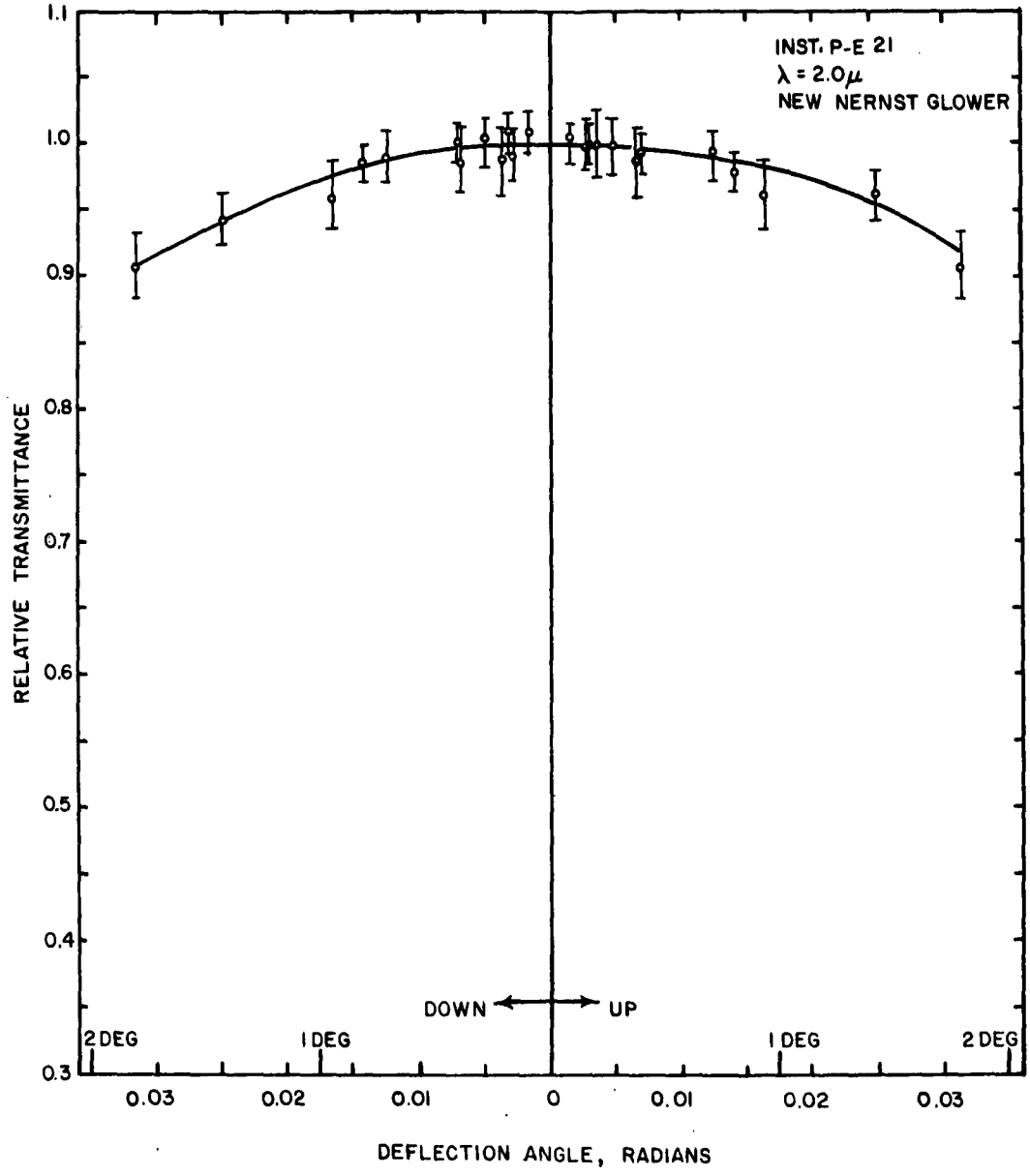

FIG. 20. Relative Transmittance Versus Deflection Angle for the Perkin-Elmer Model 21 at 2-Micron Wavelength with Newly Installed Nernst Glower, Up-Down Deflection.

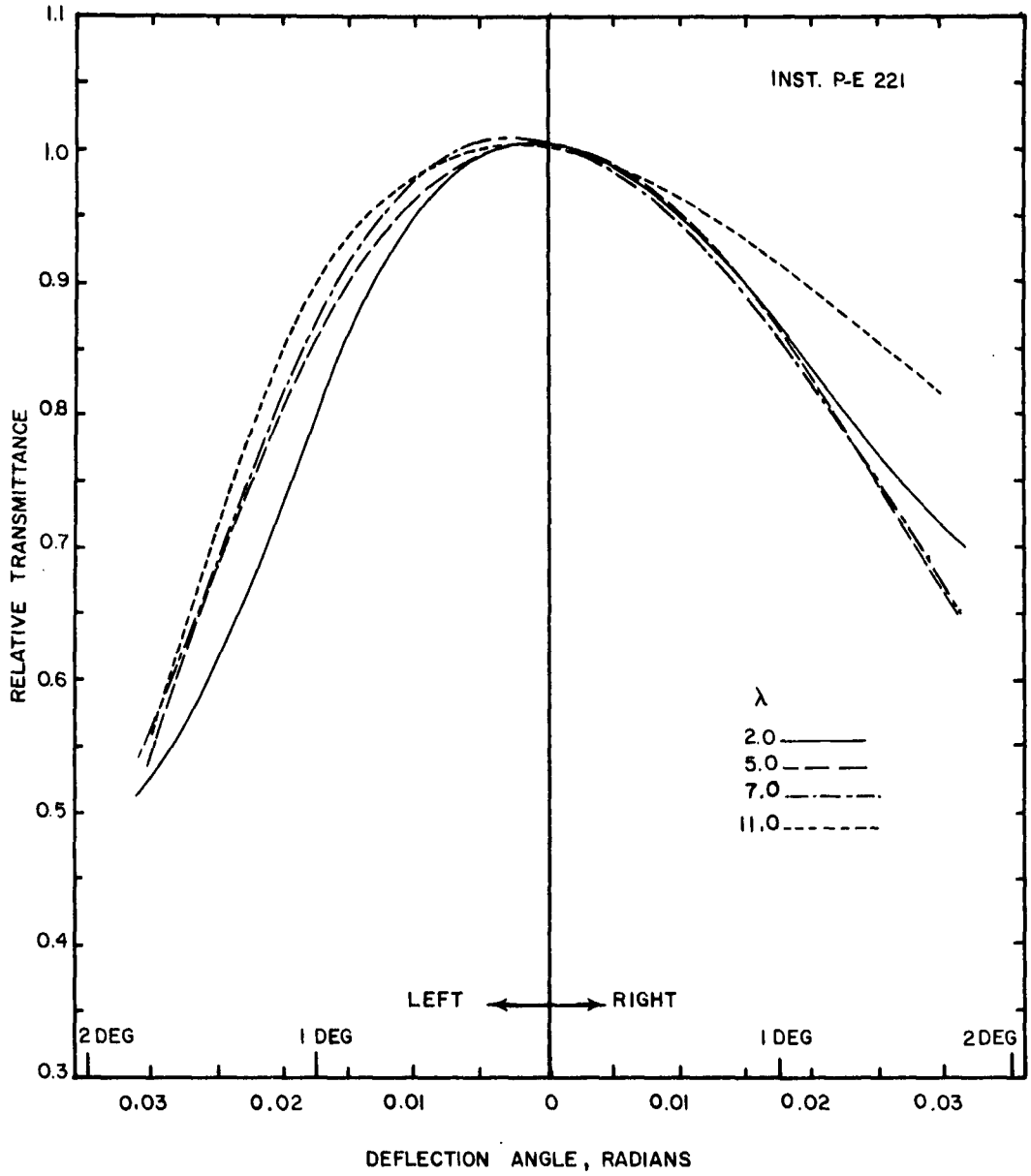

FIG. 21. Relative Transmittance Versus Deflection Angle for the Perkin-Elmer Model 221 at Several Wavelength, Left-Right Deflection.

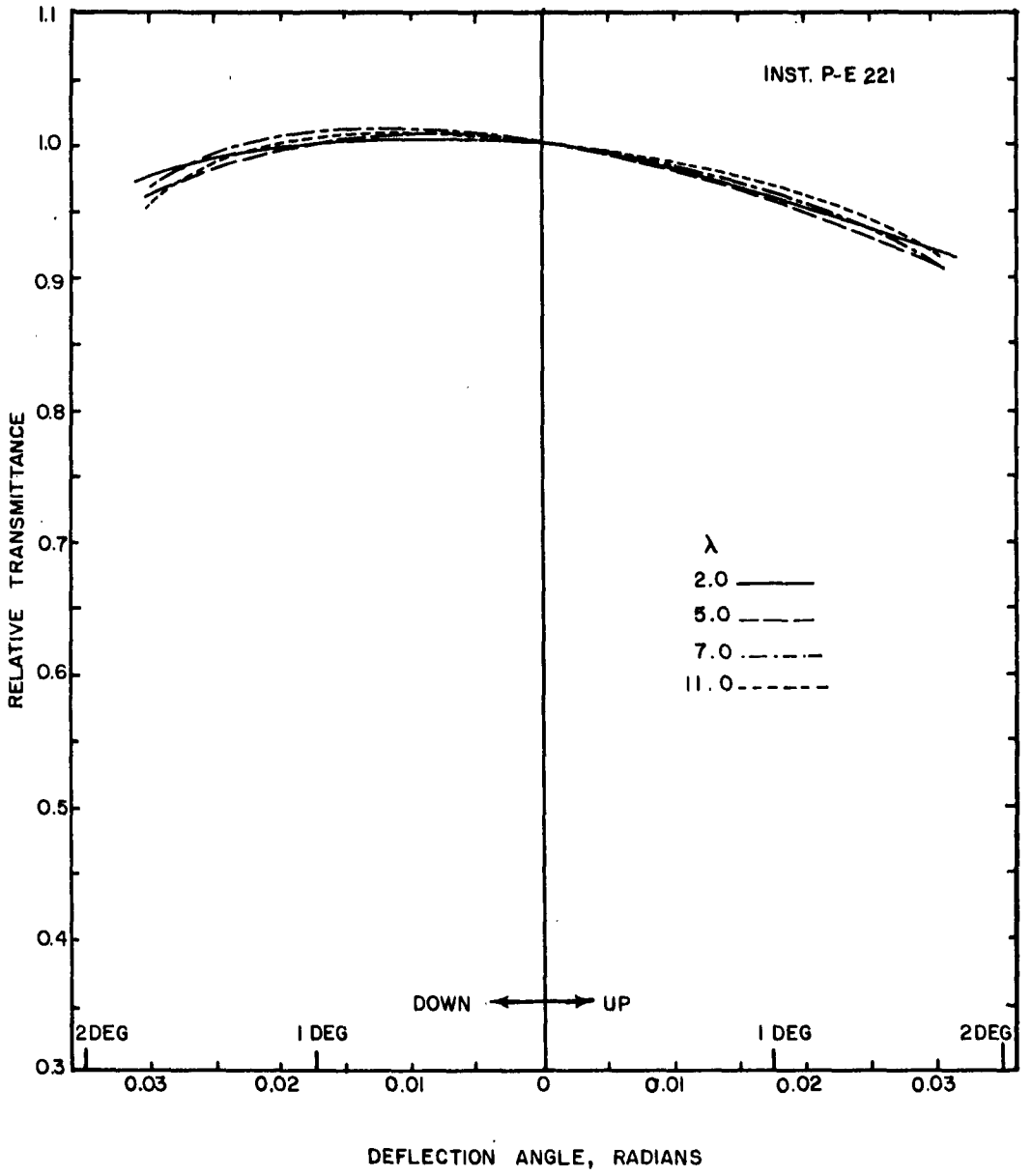

FIG. 22. Relative Transmittance Versus Deflection Angle for the Perkin-Elmer Model 221 at Several Wavelengths, Up-Down Deflection.

SUMMARY

RESULTS

Basically, the apparent change in transmittance for a deflected beam is simple optical misalignment. Figure 18 indicates that for certain conditions the measured value of transmittance can be greater with a deflected beam than with an undeflected beam. This is a consequence of the instrument not being exactly aligned for maximum energy. It is more important to have the instrument aligned whereby the energies of the sample and the reference beams are identically proportional throughout the wavelength region of the instrument than it is to have the instrument aligned for maximum energy. The sample beam energy is changed or lost in three ways: (1) the source image is displaced from the entrance slit of the monochromator, (2) a portion of the beam is greatly deflected by multiple internal reflections within the sample, and (3) part of the beam is deflected outside the mirrors which guide it into and through the monochromator.

Figures 19 and 20 are labeled "New Nernst Glower" to differentiate them from the other curves taken from the Perkin-Elmer Model 21 data. They differ only in that a new source was installed in the instrument. No change was made in the alignment of the optics. Comparison of Fig. 13 and 19 reveals that the shape of the curve was maintained but was displaced to the left for the new Nernst glower. This suggests that the new glower was not placed in the exact position of the old source. These comparisons indicate that optical alignment is critical and can make the reproducibility of information from these instruments more sensitive to nonparallel-faced material.

The curves of relative transmittance and of the transmittance against specimen rotation for the Perkin-Elmer Models 21 and 221 are, roughly, alike. Those for the Cary Model 14 differ somewhat from the equivalent Perkin-Elmer plots. This variation is mostly caused by the difference in optical systems. The spectrophotometers are more sensitive to horizontal beam deflections than to vertical. This difference is attributed to the fact that the slits in the instruments are vertical.

It is important to observe that the reproducibility of transmittance measurements is a critical function of refraction index. Quartz, with a low refractive index, did not produce a detectable error with a 10-mil wedge; however, germanium, with a high refractive index, showed a sizeable error with the 1-mil wedge.

APPLICATION

Specific applications can be made of the results of this study. If one wishes to obtain a slab of material to be used as a window for an infrared instrument and the transmittance is to be measured on a spectrophotometer comparable to those described herein, then the maximum allowable tolerance on the parallelism of the sides can be established to guarantee a given accuracy and reproducibility of transmittance measurements from the spectrophotometer. It is significant to note that these tolerances are nearly independent of wavelength.

Figure 23 is a reconstruction of the left deflection in Fig. 17 for the Cary 14 spectrophotometer. This portion was chosen for reconstruction because it represents the most critical instrument investigated. For this error graph, the deflection angle has been replaced by its independent variables, refractive index, and wedge angle. Relative transmittance has been expressed as percent error in transmittance. In Fig. 23 the error, or irreproducibility in transmittance, is clearly shown for its dependence on refractive index and wedge angle.

The following specific example illustrates an application of the findings of this study. Assume that it is desired to know the transmittance of an indium antimonide slab to within 10%. The refractive index of indium antimonide is approximately 3.5. From Fig. 23 the maximum allowable wedge is 0.0028 radian. Hence, the indium antimonide window must be fabricated with sides parallel to within ± 0.0028 radian to guarantee that the transmittance value obtained from the spectrophotometer will be within 10% of the true transmittance. In terms of difference in thickness across a 1-inch diameter disk, the tolerance is ± 0.0028 inches. If the transmittance is to be found with a reproducibility of less than 10%, then the restrictions on the wedge tolerance must be accordingly tightened.

In the even that one has a slab, or window, of an infrared optical material that already has more than the desired tolerance in wedge, then the true transmittance can be approached by orienting the slab in the sample beam to give maximum transmittance. This statement is verified by Fig. 8, 9, and 10 which show the variation with rotation.

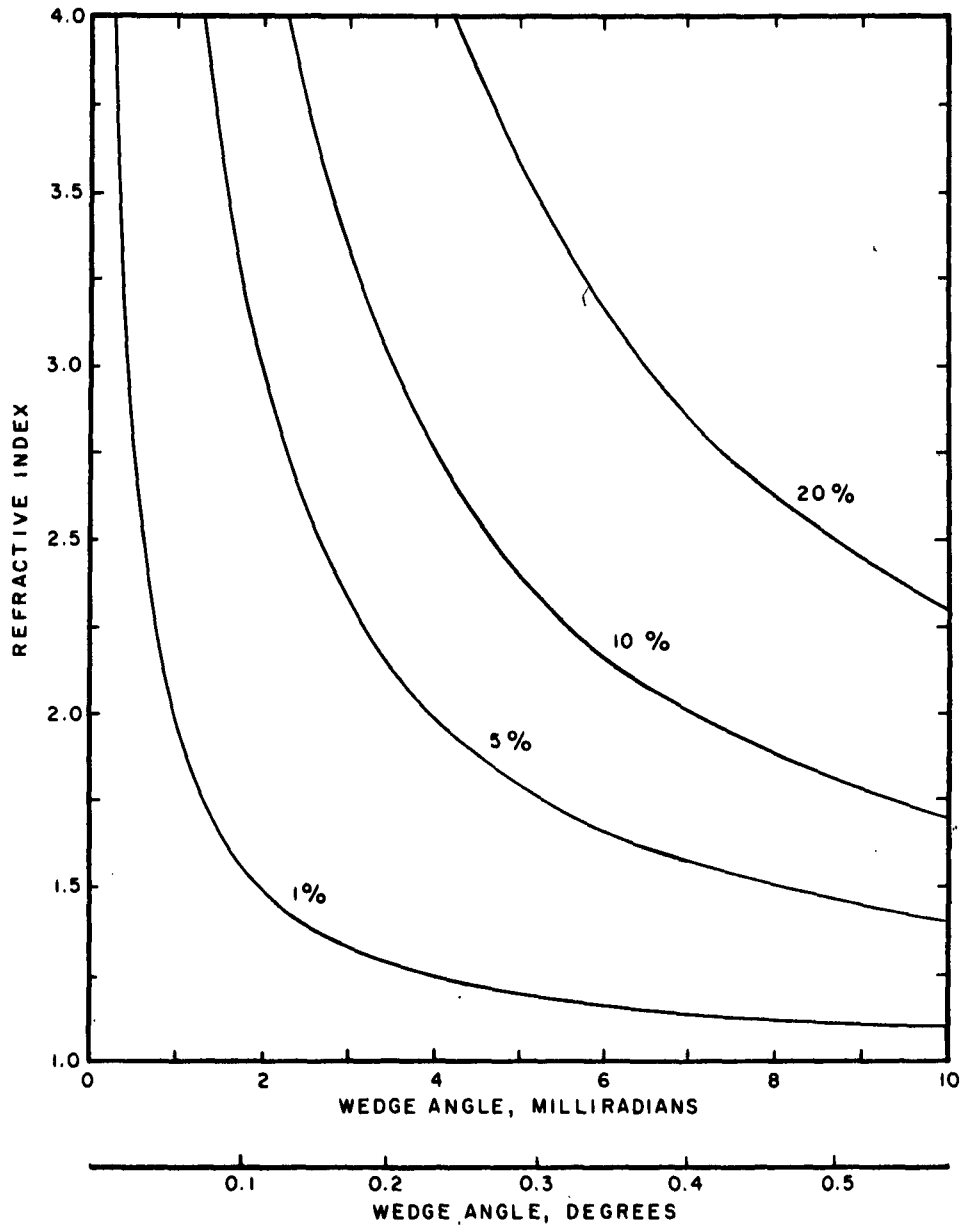

FIG. 23. The Dependence of Percent Error on Refractive Index and Wedge Angle.

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ABSTRACT CARD

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Transmittance Measurements of Optical Materials As Affected by Wedge Angle and Refractive Index in the 2- to 15-Micron Range, by K. B. LaBaw, A. L. Olsen, and L. W. Nichols. China Lake, Calif., NOTS, April 1963. 32 pp. (NAVWEPS Report 8086, NOTS TP 3116), UNCLASSIFIED.

ABSTRACT. Error in the measurement of transmittance can be made with samples having nonparallel faces. This has been investigated because of the extensive use of transmittance as a tool for determining properties of semitransparent materials. Specimens of fuzed quartz, arsenic trisulfide, silicon,

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